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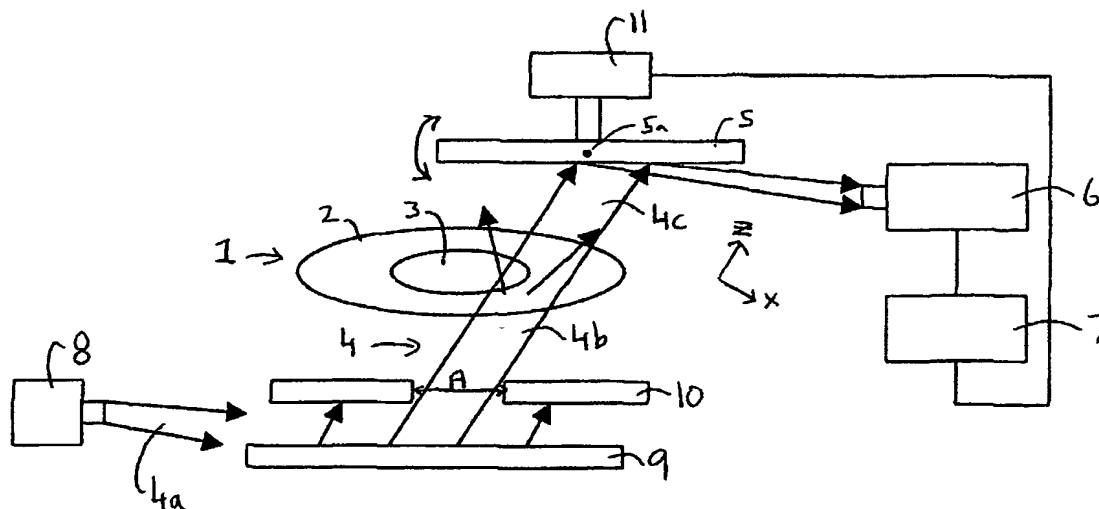
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(54) Title: METHODS AND APPARATUS OF SAMPLE ANALYSIS



(57) Abstract: An x-ray beam (4) from a rotating anode source (8) is passed through an object (1) via a monochromator (9) and slit member (10) in order to determine the object's internal structure. The emerging radiation that is within the acceptance angle of a crystal analyser (5) is diffracted onto a PIN diode detector (6), which records an intensity profile of the radiation detected as a function of angular position of the crystal analyser (5). The resulting profile is analysed to provide a complex refractive index profile for the object (1) across the width of the beam (4). The analysis method and apparatus utilises both absorption and refraction information, and can provide both qualitative and quantitative information on the object's structure, with the dimensions of the slit member (10) providing an analytical intensity profile.

Methods and Apparatus of Sample Analysis

The present invention relates to methods and apparatus for the analysis of a sample. It relates particularly to x-ray analysis, and has applications in both the medical and non-medical fields. It may be used in the analysis of organic and inorganic substances, and crystalline and amorphous materials.

X-ray analysis in the form of x-ray radiography is well-known in the medical field, and is a widely used technique for medical imaging.

In its simplest form, a radiographic device comprises an x-ray source and an x-ray sensitive film arranged opposite to one another on either side of a patient. X-rays from the source are directed at the patient's body, and those that pass through are recorded on the film. Since bone and soft tissue absorb different amounts of radiation, an image of lighter and darker areas is captured on the film, and can be used to identify internal structure of the patient.

A problem with this technique is that organic materials are generally weak absorbers of X-ray radiation, and so distinguishing between different types of soft tissue can be difficult. For example, it can be difficult to distinguish between healthy soft tissue and a tumour, especially in a tumour's early stages.

X-ray techniques based on phase imaging have recently been promoted for overcoming this problem. Rather than use x-ray absorption, these methods attempt to provide better image contrast by utilising instead the phase shift of x-rays as they pass through regions of the sample of different refractive index. Phase imaging techniques can provide improvement over radiographic techniques, and can allow for better definition of soft tissue.

A problem with both radiographic and phase imaging techniques is that in order to use relatively short exposure times, they generally require the incident radiation to be of a relatively high intensity. Damage of the object under examination by the x-rays is therefore of concern. This is especially so in medical imaging, where strict protocols must be observed as to radiation dosages and the like.

A further point of note is that it can be difficult and expensive to obtain high resolution in both techniques. For example, even the use of the most expensive CCD cameras (CCD refers to charge couple device) on the market today still imposes undesirable limits on image resolution and dynamic range.

The present invention aims to provide an alternative x-ray analysis technique that, in its various aspects and embodiments, can provide a number of advantages both in the medical and non-medical fields.

Viewed from a first aspect, the present invention provides a method of analysis of an object, the method including the steps of:

generating non-planar penetrating radiation;
diffracting the radiation from a monochromator to provide a beam of monochromatic penetrating radiation;
irradiating a portion of the object with the beam;
diffracting radiation that passes through the object onto a detector from an analyser;
rotating the analyser through a plurality of angular positions; and
measuring the intensity of the radiation incident on the detector as a function of analyser position.

Viewed from a second aspect, the present invention provides apparatus for the analysis of an object, the apparatus including:

a source of non-planar penetrating radiation;
a monochromator for diffracting the non-planar penetrating radiation to provide a beam of monochromatic penetrating radiation;

a detector for detecting radiation that passes through the object;
an analyser for diffracting radiation that passes through the object onto the detector;

means for rotating the analyser between a plurality of angular positions;
and

means for recording the intensity of the radiation incident on the detector as a function of analyser position.

Each intensity value detected through the present invention (i.e. for each angular position of the analyser) corresponds to the intensity of the x-ray photons that are diffracted by the analyser onto the detector from across the whole of the radiation beam that has penetrated the sample object. The resultant intensity profile includes information as to both the absorption and refraction of x-rays by the sample, and facilitates the determination of for example the shape and structure of the analysed portion in a single experiment.

As is preferred, the present invention allows for the determination of a complex scattering function of the analysed portion from the intensity measurements. This can provide the complex refractive index of the portion of the object under analysis as a function of cross-section transverse to the direction of propagation of the beam.

The invention contrasts with the prior art radiographic and phase contrast techniques that generally view the object under analysis either in a purely absorptive or in a purely phase manner.

The present invention is further distinct from the prior art in that it can be thought of as obtaining and analysing information in "reciprocal space" rather than in "real space".

The present invention facilitates the analysis of an object at high resolutions in an inexpensive manner. For example, there is no need to use an expensive high resolution CCD detector, as the detector used in the present technique need not provide spatial resolution (spatial resolution is instead provided by the rotation of the analyser, and may be determined by the full aperture of angular rotation).

The present technique can provide higher resolution than the prior art, and may for example provide resolutions in the region of 0.05 – 0.5 nm.

The present invention is also able to efficiently utilise the x-ray photons emerging from the portion of the object analysed, since it effectively integrates the emerging x-rays across the whole width of the analysing beam, and the detector may start counting from a single photon of light. The present invention thus facilitates the use of x-ray radiation of lower intensity, and may provide better data statistics and so analysis quality than imaging techniques.

It should be noted that the present invention analyses non-Bragg diffracted x-rays exiting from the sample object, and thus is distinct from e.g. standard crystallography and the like.

Generally, an analysis of non-Bragg diffracted waves would be considered of no use, as it was not thought possible to obtain any meaningful information from such an analysis. Indeed, the idea of obtaining diffraction effects at all for non-periodic structures, such as amorphous materials was not entertained.

The inventor of the present analysis apparatus and method has not only found that meaningful information can be obtained from a study of non-Bragg diffracted waves from both crystalline and non-crystalline samples, but has also found that quantitative information is achievable.

5 Further, not only has the inventor found this, but also has determined contrary to what might be thought, that such information is obtainable even when using a standard laboratory source (as opposed for example to when using beams of ultra-high intensity and ultra-low divergent radiation that are only found in the most expensive of synchrotrons).

10 The inventor has thus been able to provide an analysis technique that offers a practicable and high-resolution alternative to the prior art imaging methods.

The use of laboratory sources (as opposed to top-end e.g. 1 kilometre long synchrotron radiation sources) may be characterised by the generation of
15 non-planar penetrating radiation, which is then collimated by the monochromator (with a resultant loss in beam intensity). Non-planar penetrating radiation in the present application may be considered to be radiation having an angular divergence of greater than 10 seconds of arc when incident at the monochromator.

20 In a preferred embodiment, the x-ray source is a characteristic line source such as an x-ray tube. In such sources, material is bombarded by electrons to generate the x-rays. The material may be e.g. copper, molybdenum, silver or tungsten.

In an especially preferred embodiment, the source is a rotating anode
25 source. Such sources are similar to standard x-ray tube sources, but the material bombarded is rotated. Rotating anode sources are able to provide relatively high intensity beams so as to provide greater resolution and better signal to noise ratios in the intensity data.

For comparison, an x-ray tube typically generates x-rays having an
30 energy range of from about 5 to 150 keV and having an intensity of about 10^6 photons/sec, whilst a rotating anode source typically generates x-rays having an energy range of from about 5 to 60 keV and having an intensity of about 10^8 photons/sec. Synchrotron radiation typically has an energy range of from about 1 to 100 keV and an intensity of about 10^{12-18} photons/sec.

Other laboratory sources of x-rays could also be used. These could include for example small-scale synchrotrons, e.g. which are able to fit into a room of a building, but which, because of their small beam lengths, may still require optical elements, such as a monochromator, to provide a beam of low angular divergence.

The analyser may take any suitable form, and for example may comprise a crystal oriented so that only x-rays of a set angle of incidence or range of angles and desired wavelength are diffracted towards the detector. It may comprise a single or double crystal that may be made of e.g. silicon or germanium. Silicon is preferred, and provides a stable and effective analyser.

The crystal analyser is the analyser of choice, but alternatives are possible. For example, the analyser could be in the form of an x-ray mirror.

The monochromator may also take any suitable form, and may take a form similar to that of the crystal analyser, e.g. a single or double crystal of silicon or germanium, or an x-ray mirror.

The monochromator and analyser pair provides the system with a set optical resolution, determined from e.g. the angular divergence of the beam leaving the monochromator and e.g. the angular acceptance of the analyser. The optical resolution will generally be the angular divergence of the beam after passing through the monochromator, or the angular acceptance of the analyzer, whichever is greater.

It is preferred that the monochromator and the analyser are of the same type and have equivalent angular divergence and angular acceptance values. However this is not essential for the operation of the invention.

The actual optical resolution of the apparatus can be taken from a rocking curve of the apparatus, i.e. an intensity profile of detected intensity versus analyser position for the apparatus without a sample in place. The optical resolution may be defined by the Full Width at Half Maximum of the rocking curve (FWHM).

The monochromator and analyser pair may be arranged so that the analyser is at its zero angle position when it is diffracting x-rays into the detector at the Bragg angle.

Preferably, x-rays pass through a slit prior to their incidence on the object, the slit size A (in a direction transverse to the direction of propagation of

the beam and in the direction in which the refractive index is being measured) being such that:

$$A \leq \lambda / \delta\theta,$$

where λ is the wavelength of the incident radiation, and $\delta\theta$ is the optical resolution of the apparatus used (e.g. the monochromator/analyser pair resolution, e.g. determined from the FWHM of the rocking curve).

A point to note here is that the optical resolution $\delta\theta$ is determined by the apparatus, rather than being a quality of the beam itself (which would be the case if a top-end synchrotron beam were used).

This use of a slit width within the above constraint ensures that the intensity profile detected is able to provide analytical functions for determining the object's refractive index, and so allows a quantitative analysis of the cross-sectional refractive index.

Typical slits widths may range from e.g. 50 to about 500 microns. A copper x-ray tube may for example utilise slit widths of between about 50 and about 300 microns, whilst a molybdenum source may utilise slit widths between about 50 and about 500 microns, and a silver source slit widths of between about 50 and about 300 microns. Preferably, the slit width is greater than 50 microns, and more preferably greater than 100 microns. It is preferably less than 500 microns, and may for example fall within the range of about 250 to about 300 microns.

The slit member that defines the slit is preferably made from tantalum, and the edges of the slit are preferably flat and parallel to the direction of propagation of the beam. This avoids the possibility that x-rays will penetrate the slit member about the edges of the slit, and cause undesired diffraction effects in the measured intensity profile.

As well as providing quantitative information, the intensity profile may merely be plotted e.g. as Log(intensity) against angular position of the analyser, and the resulting plot may be reviewed qualitatively e.g. against a similar plot for a reference sample.

The size of the slit in the second transverse direction may take any suitable value, and may be e.g. between about 0.1 mm and about 10 mm. The particular size may be dependent on the sample under analysis, and any

desired spatial resolution in the second transverse direction (e.g. if an intensity profile were to be obtained in this direction also).

Preferably the crystal analyser is rotated in a plurality of incremental steps, each step being through an angle α :

5
$$\alpha \leq \delta\theta/2$$

where $\delta\theta$ is again the optical resolution of the apparatus.

This constraint allows the complete determination of the complex scattering function. It takes into account the need to measure $2N$ points of a real function (i.e. intensity) in order to obtain N points of a complex function (which has both real and imaginary components).

A typical step may be e.g. 0.05 or 0.01 arc.secs, although other step sizes are equally possible.

The detector may take any suitable form, and, as said, need not provide any spatial resolution. Preferably, the detector comprises a PIN (P-intrinsic-N) diode detector. PIN diode detectors are able to provide a linear response over a large dynamic range. This can be an advantage in the present technique, as the scattered intensity profile to be recorded typically has a large variation in intensity, with useful information being contained in both high and low intensity areas: A typical profile of the logarithm of intensity against analyser angle comprises a large central peak with small but significant variations in intensity in the tails of the peak. As an alternative, a scintillation counter or the like may be used.

Preferably, a complex scattering amplitude of the irradiated portion of the object (in reciprocal space) is calculated from the detected intensities, and a complex scattering function of the irradiated portion is determined by taking an inverse Fourier Transform of the complex scattering amplitude.

The method preferably includes the steps of: normalising the detected intensities; calculating the modulus of the complex scattering amplitude from the normalised intensity; calculating phase information (e.g. a minimal phase) of the complex scattering amplitude from the modulus of the complex scattering amplitude; and determining the complex scattering amplitude from the modulus and the phase information.

The analysis of the detected intensity profiles may be carried out by any suitable means, and may be implemented in software running on any suitable

computing apparatus, for example a personal computer, as would be well understood by a person skilled in the art. The computing apparatus could control the whole analysis operation and co-ordinate control of the analyser and detector, e.g. control the rotation of the analyser and the recording of the
5 detected intensities against angular position of the analyser.

The sample may be positioned relative to the slit, such that significant variation in the sample refractive index is expected only in the diffraction plane of the monochromator-analyser pair.

In one preferred embodiment, the object is translated relative to the
10 beam, e.g. in a linear fashion, between obtaining profiles, so as to provide an analysis of the object over a number of beam widths. The results of the separate analyses of the various portions of the object may then be combined so as to provide a complex refractive index profile of the object over a large cross-sectional area. An object can therefore be mapped over a large area at a
15 high resolution.

The present invention provides a refractive index profile for the sampled portion of the object in a direction transverse to the direction of beam propagation, the resulting profile also being the refractive index integrated over the distance that the beam propagates through the sample. In order to provide
20 three-dimensional information, tomographic techniques may be employed.

It should be noted that the restriction of the slit width in accordance with the constraint discussed above is in itself an important feature of the present invention, and provides an intensity profile that is analytical and so allows for a quantitative analysis of the refractive index of the sample.

25 Accordingly, viewed from a further aspect, the present invention provides a method of analysis of an object, the method including the steps of:

generating penetrating radiation;

diffracting the radiation from a monochromator to provide a beam of monochromatic penetrating radiation;

30 passing the beam of radiation through a slit, the slit size A (in a direction transverse to the direction of propagation of the beam) being calculated such that:

$$A \leq \lambda/\delta\theta$$

where λ is the wavelength of the incident radiation, and $\delta\theta$ is the optical resolution of the apparatus used in the method;

irradiating a portion of the object with the beam;

diffracting radiation that passes through the object onto a detector from

5 an analyser;

rotating the analyser through a plurality of angular positions; and

measuring the intensity of the radiation incident on the detector as a function of analyser position.

The present invention may also be seen as providing an apparatus for
10 the analysis of an object, the apparatus including:

a source of penetrating radiation;

a monochromator for diffracting the penetrating radiation to provide a beam of monochromatic penetrating radiation;

a slit member defining a slit through which the beam passes prior to the
15 beam's incidence on the object, the slit size A (in a direction transverse to the direction of propagation of the beam) being calculated such that:

$$A \leq \lambda/\delta\theta$$

where λ is the wavelength of the incident radiation, and $\delta\theta$ is the optical resolution of the apparatus;

20 a detector for detecting radiation that passes through the object;

an analyser for diffracting radiation that passes through the object onto the detector;

means for rotating the analyser between a plurality of angular positions; and

25 means for recording the intensity of the radiation incident on the detector as a function of analyser position.

Viewed from a still further aspect, the present invention provides a method of analysis of an object, the method including the steps of:

irradiating a portion of the object with a beam of monochromatic x-rays;

30 detecting the intensity profile of an angular spectrum of the x-rays emerging from the irradiated portion; and

determining a complex scattering function for the irradiated portion of the object under analysis.

Viewed from another aspect, the present invention provides a method of analysis of an object, the method including the steps of:

irradiating a portion of the object with a beam of monochromatic x-ray radiation;

5 diffracting x-rays emerging from the sample into an x-ray detector using an analyser means; and

obtaining an angular spectrum of non-Bragg diffracted x-ray intensities as a function of angular position of the analyser means.

10 Viewed from a further aspect, the present invention provides a method of analysis of an object, the method including the step of collecting generic (non-Bragg diffracted) x-ray diffraction data from a portion of the object and analysing the data to obtain a complex refractive index of the sampled portion in a direction transverse to the beam propagation.

15 These further aspects may extend to apparatus for applying the methods, and may include any of the features of the preceding aspects of the present invention.

20 Embodiments of the present invention will now be described, by way of example only, with reference to the accompanying drawings. It is to be understood that the particularity of the drawings does not supersede the generality of the preceding description of the present invention.

In the drawings:

Figure 1 is a schematic diagram of x-ray analysis apparatus in accordance with a first embodiment of the present invention;

25 Figure 2 shows scattered intensity profiles for three analysed samples as a graph of Log(intensity) against detection angle;

Figure 3 shows the scattered intensity profile for a sample when placed at two different positions relative to a beam slit; and

Figure 4 represents the profile of the real component of the complex refractive index for the two profiles of Fig. 3.

30 Referring to Fig. 1, an object 1 to be analysed, e.g. a blood vessel, will have regions of differing refractive index, e.g. an outer region 2 consisting of the blood vessel wall, and an inner region 3 consisting of the blood itself.

Overall, in order to analyse the internal structure of the object 1, a beam of x-ray radiation (generally labelled 4) is passed through the object 1, and the

emerging radiation that is within the acceptance angle of a crystal analyser 5 is diffracted onto an x-ray detector 6. The detector 6 records an intensity profile of this radiation as a function of angular position of the crystal analyser 5, and suitable control means 7, such as a computer running suitable software, analyses the resulting profile to provide a complex refractive index profile for the object 1 across the width of the beam 4 in the x-axis direction (transverse to the direction of propagation of the beam 4).

In more detail, in the apparatus shown, an x-ray source 8, such as an Mo x-ray tube with $K_{\alpha} = 0.709\text{nm}$, generates an x-ray beam 4a that is diffracted from a crystal monochromator 9 and passed through a slit member 10 so as to provide a narrow collimated beam of low divergence monochromatic x-rays 4b that is incident on the sample object 1.

The monochromator 6 is an Si crystal providing a (333) asymmetric reflection, and the crystal is cut such that (111) atomic planes are at 18.5° to the surface.

The slit member 10 is made of e.g. tantalum and the sides of the slit are flat and parallel to the direction of propagation of the beam so as to prevent x-rays from passing through the edges of the slit member 10 about the slit opening and causing undesirable diffraction effects.

The slit size, A , is selected to satisfy the relationship:

$$A \leq \lambda/\delta\theta$$

where λ is the wavelength of the incident radiation, and $\delta\theta$ is the optical resolution of the apparatus as determined by e.g. the angular divergence of the beam after the monochromator or the angular acceptance of the analyzer. In the present case, the optical resolution may be determined by the Full Width at Half Maximum (FWHM) of the rocking curve of the slit member 10, the rocking curve being the intensity profile obtained by the apparatus in the absence of a sample to be analysed.

The x-ray beam 4b is absorbed and scattered in its passage through the sample 1, and the emerging x-rays 4c that fall within the acceptance angle of the crystal analyser 5 are diffracted towards the detector 6.

The crystal analyser 5 may be of the same configuration as the monochromator 9, and may comprise an Si crystal providing a (333)

asymmetric refraction, the crystal being cut such that (111) atomic planes are at 18.5° to the surface.

The crystal analyser 5 is arranged opposite to the entry point of the x-rays into the object 1, and is mounted to rotate in incremental steps about an axis 5a over a set range of angular positions so as to present itself at a plurality of different angles to the x-ray beam 4.

The means 11 for rotating the crystal analyser 5 may comprise e.g. a goniometer, and may be controlled by the control means 7, which also records the intensity of x-rays received at the detector 6 as a function of the angle of the analyser 5 from its zero position. The "zero" position of the analyser 5 relative to the beam 4 corresponds to the Bragg reflection angle of the analyser crystal, and in this position, the analyser is in this embodiment substantially parallel to the monochromator 9.

In use, the crystal analyser 5 is rotated between e.g. -30 arc.secs and +30 arc.secs of the Bragg reflection angle, in steps of e.g. 0.05 arc.secs. The steps should be less than half the optical resolution of the apparatus, e.g. as defined by the monochromator and analyser pair (This takes into account the need to acquire 2N real values in order to compute N complex values).

At each step, an intensity reading is recorded. This may be for a set period of time, for instance between about 1 to about 10 seconds. Alternatively, the intensity of the radiation at each step may be measured until a set number of photons is reached, e.g. 1000 photons. In such a case it may take e.g. from about 10 to 20 seconds to measure an intensity value at an extreme angular position, and e.g. from about 0.1 to 0.5 seconds to measure intensity at a point close to the peak of the profile. Each resulting intensity value may be modified to take account of the time taken to obtain it, e.g. by dividing the intensity value by the measurement time.

The detector 6 may be a PIN diode detector, which is able to provide a linear response over a large dynamic range. This is useful for the present technique, as the detector 6 needs to record both a large central peak of substantially unabsorbed and unrefracted radiation, as well as meaningful small perturbations in the tails of the profile caused by phase shifts in the x-rays as they pass through regions of the object of different refractive index. Alternative detectors may also be used, e.g. a scintillation counter.

Examples of intensity profiles that may be obtained through the use of the apparatus of Fig. 1 are shown in Fig.2, which plots Log(Intensity) against crystal analyser angle.

The profiles A-C are of a polyethylene tube (approximately 6 mm in diameter and sealed at both ends) filled with air, water and butter respectively, so as to roughly simulate a blood vessel configuration: The air is used to imitate an empty blood vessel, whilst the water and butter are used to imitate a blood vessel filled with clear blood and organic fat respectively.

The profile RC provides an analyser rocking curve corresponding to no sample. The rocking curve may be used to calculate the optical resolution of the apparatus by determining the FWHM.

It should be noted that the scans are offset by about 1 decade for clarity, and that the sample may be immersed in water to reduce the change in refractive index that would otherwise occur at the interface of the sample object with air.

Inspection of such plots can provide qualitative information as to the object under analysis, e.g. one may be able to tell from such a plot if a blood vessel is abnormal.

The present invention can however also provide quantitative information as to the structure of the object from a suitable analysis of the intensity profile.

Overall, the concept is to obtain (in reciprocal space) the complex diffraction amplitude of the portion of the object analysed by the x-ray beam, and from this to obtain the complex refractive index profile of the analysed portion across the width of the beam. This complex refractive index will have information on both the absorptive and refractive features of the object under analysis, and so will provide information on the structure of the analysed portion of the object.

The analysis relies on the analytical properties of the complex diffraction amplitude, which is provided by the use of a slit width A within the above-noted constraints.

The refractive index profile obtained will be that along the x-axis of the portion of the object that is analysed (integrated over the length of the object through which the beam passes), and assumes that the object is homogeneous

or has a slowly varying refractive index in the y-axis direction (In Fig. 1, this direction is out of the plane of the paper).

As a specific example, a profile may be analysed by firstly normalising the measured intensity, $I(\theta)$, by the square modulus of the complex scattering vector:

$$Q^2 = q^2 + \mu^2,$$

where $q = \theta/\lambda$ and μ is the linear attenuation coefficient of the material.

The normalised intensity is:

$$\tilde{I}(Q) = I(Q) \times Q^2.$$

Next, the modulus $|R(Q)|$ of the complex diffraction amplitude, $R(Q)$, is calculated as the square root of the normalized intensity:

$$R(Q) = \sqrt{\tilde{I}(Q)}.$$

The minimal phase is then calculated from the square root of the measured intensity using a logarithmic Hilbert transform. This can be implemented using the relation between Hilbert and Fourier transforms (see e.g. R.N. Bracewell, The Fourier transform and its applications (McGraw-Hill, New York, 1986):

$$\varphi^{\min}(Q) = -\frac{1}{\pi} P \int_{-\infty}^{\infty} \frac{\ln|R(q)|}{q' - q} dq',$$

where P is the Cauchy principle value of the integral.

The solution for the Complex Diffraction Amplitude can then be synthesised as:

$$R(Q) = |R(Q)| \exp\{i\varphi^{\min}(Q)\}.$$

Next, the inverse Fourier transform of $R(Q)$ is taken:

$$\chi(x) = \mathcal{F}^{-1}\{R(Q)\}.$$

The complex refractive index profile of the sample across the width of the beam can then be obtained by taking the integral:

$$\chi(x) = \int_0^A \chi(x) dx.$$

An unambiguous solution to the complex refraction index profile may be obtained either by using intensity profiles collected for two different x-ray

energies or by using a single intensity profile compared to previously obtained benchmark profiles for similar samples. The results obtained for the two profiles can then be compared so as to identify physically real roots from artefacts of the computation.

5 In one experiment, the apparatus of Fig. 1 was used to analyse a sample of nylon ($C_6H_{13}NO_2$, density – 0.4336 g/cm^3) of 80 micron diameter.

The sample was arranged to occlude the slit in the slit member 10 such that the whole sample is “seen” through the slit plus a bit of air.

The apparatus had an optical resolution of $\delta\theta = 0.12 \text{ arc.sec.} = 5.87 \times 10^{-7}$ rad, and the slit aperture was selected to be 110 microns, which satisfies the condition that the slit aperture must be smaller than:

$$A = \lambda / \delta\theta = 7.09 \times 10^{-11} / 5.87 \times 10^{-7} \cong 120 \text{ micron.}$$

The angular step in the diffraction pattern, i.e. the angular step of the crystal analyser 5 was chosen to be 0.05 arc.sec. – at least two times smaller than the optical resolution.

The intensity profile (generic optical diffraction pattern), $I(\theta)$, was determined from the sample by the angular scanning of the analyser from -10 arc.sec. to $+10 \text{ arc.sec.}$ (giving 401 experimental points). This gave a total angular scan aperture:

$$\theta_{\text{aperture}} = 20 \text{ arc. sec.} = 9.7 \times 10^{-5} \text{ rad.}$$

This corresponds to a resolution for the reconstructed profile of the refractive index of the sample across the beam width of:

$$\Delta x = \lambda / \theta = 7 \times 10^{-11} / 9.7 \times 10^{-5} = 0.72 \text{ micron.}$$

Further examples of intensity profiles obtained using the apparatus of Fig. 1 are shown in Fig. 3. They are for a model blood vessel comprised of a 1 mm diameter grass straw cylinder located partially in the x-ray beam 4 (only one edge of the straw was within the beam, the other edge was blocked by the slit member 10).

The sample was immersed in water, so that the total thickness of the sample cell was about 1.5 mm, with $2 \times 50 \text{ micron Kapton}^{\text{TM}}$ windows to the sample cell.

The x-ray radiation was $\text{Mo K}_{\alpha 1} = 17.5 \text{ keV}$.

The monochromator/analyser pair used a single silicon dislocation-free crystal with an asymmetric cut that had an asymmetry factor $b = 0.038$. Bragg reflection was Si 333, and Darwin's width was 0.12 arc.sec.

The slit size was 0.1 mm (x-axis, A dimension) x 5 mm (y-axis).

5 The two profiles A' and B' shown in Fig. 3 are for two positions of the sample relative to the 100 μm wide slit. Profile RC' is a rocking curve for the apparatus corresponding to no sample, i.e. an open slit.

In scan A', the sample occludes the slit by $58 \pm 2 \mu\text{m}$, and in scan B', the sample occludes the slit by $40 \pm 2 \mu\text{m}$.

10 Fig. 4 shows the reconstructed profile of the real component of the complex refractive index of the sample (proportional to the thickness/density profile in the direction of the incident x-rays).

The drop on the right side of the profile corresponds to the slit edge, and the drop on the left-side corresponds to the edge of the sample. The left-side
15 drop has shifted by about 15 micron between the two profiles, corresponding to the movement of the sample between measurements of the profiles.

The spatial resolution of the reconstruction was 1.5 μm .

The basis for the present approach to x-ray analysis is as follows. In the case of a non-crystalline sample, the complex scattering function of the sample
20 may be expressed as:

$$t(x) = \exp\left\{\frac{2\pi}{\lambda}i \int n(x, y, z') dz'\right\}$$

where n is the map of the complex refractive indices, $n(x, y, z) = 1 - \delta(x, y, z) - i\beta(x, y, z)$, and z is the direction of the incident wave propagation, for an ideally monochromatic source of wavelength λ .

25 The experimentally measured scattered intensity (i.e. that obtained at detector 6) is the square of the modulus of the complex scattering amplitude, $I = |T(Q)|^2$, where $T(Q)$ is given as the Fourier transform of the complex scattering function $t(x)$:

$$T(Q) = |T(Q)| \exp(i\phi(Q)) = \int_{\text{all space}} t(x) \exp(2\pi i Qx) dx$$

30 where $T(Q) = u(q_r, q_i) + iv(q_r, q_i)$ and q_r, q_i are the real and imaginary parts respectively of the complex scattering vector $Q = q_r + iq_i$.

The inverse problem of determining the complex scattering function $t(x)$ from the experimentally observed scattered intensity profile $I(Q)$ depends on the theoretical approach to the relation between the modulation function and the observed intensity function.

- 5 The phase $\phi(Q)$ of the experimentally observed x-ray scattering profile may be retrieved via a logarithmic dispersion relation, an approach that is valid under the kinematical theory of x-ray scattering:

$$\phi(Q) = -\frac{1}{\pi} P \int_{-\infty}^{\infty} \frac{\ln|T(Q')|}{Q' - Q} dQ' + 2 \sum_m \arg(Q - Q^m) = \phi^{\min}(Q) + \sum_m \phi^m(Q)$$

- 10 where Q^m ($m=0,1,2,\dots M-1$) are the zeros of $|T(Q)|$ in the upper half of the complex plane, and P is the Cauchy principal value of the integral. The zeros of the scattered amplitude, Q^m , are of unknown number, M , and may in principle be infinite in number. However, the analyticity of the CDA allows its unique representation as a complex polynomial function:

$$T(Q) = \prod_{k=0}^{K-1} (Q - Q^k)$$

- 15 in which the number of zeros is limited to the discrete number of points K in the experimentally collected data set.

- Once the x-ray phase, $\phi(Q)$, is determined, e.g. the minimal phase change term, an inverse Fourier transform of the calculated complex scattering amplitude $T(Q)$ can be used to find the complex scattering function $t(x)$ and
20 provide information on the thickness/composition profile.

- Overall, the present invention can provide a method and apparatus for the x-ray analysis of a sample, which is able to provide high resolution and good contrast without great expense, and can provide information as to both absorptive and refractive features of the sample in a single experiment. The
25 invention utilises an angular spectrum of non-Bragg diffracted x-rays obtained from standard laboratory x-ray sources, and is not limited for example to expensive top-end synchrotron sources or the like. The invention can ensure analyticity of the resulting profiles through the slit constraints determined with respect to the optical resolution imposed by the analysing apparatus.

- 30 The invention may be used in situations requiring non-invasive analysis techniques, and may be used in the medical and non-medical fields, in relation

to organic and non-organic materials, inanimate or living objects, and crystalline and amorphous substances.

In the medical field, the invention may be used e.g. in the cardiovascular, oncological and urological fields in measuring occlusions.

5 In non-medical fields, the present invention may be used in e.g. the characterisation of light metal alloys, e.g. as used in the aerospace industry. The invention may be used to measure e.g. stresses and strains in crystalline material (the invention analysing the movement of groups of atoms, as opposed to detecting individual atoms themselves).

10 The invention may be extended to provide further information about a sample e.g. in the y-axis and z-axis. For example, the apparatus could be arranged to further scan in the y-direction, and the object or apparatus could be rotated to provide other beam paths through the object, and to correlate the information obtained in a manner as in tomography.

15 It is to be understood that various modifications and/or alterations may be made to the above without departing from the spirit of the invention as outlined herein.

For example, although discussed in terms of x-rays generated by an x-ray tube, any other suitable x-ray source could be used, including e.g.
20 synchrotron radiation. Also, the monochromator and analyser could take any other suitable form, and could e.g. be comprised of double crystals and could be made of germanium. Alternative diffraction analysers could also be used such as X-ray mirrors. The detector could also take any other suitable form.

Claims

1. A method of analysis of an object, the method including the steps of:
generating non-planar penetrating radiation;

5 diffracting the radiation from a monochromator to provide a beam of
monochromatic penetrating radiation;

irradiating a portion of the object with the beam;

diffracting radiation that passes through the object onto a detector from
an analyser;

10 rotating the analyser through a plurality of angular positions; and
measuring the intensity of the radiation incident on the detector as a
function of analyser position.

2. The method of claim 1, including the step of determining a complex
15 scattering function of the portion of the object under analysis from the intensity
measurements.

3. The method of claim 1 or 2, including the step of passing the beam of
radiation through a slit prior to the beam's incidence on the object, the slit size A
20 in a direction transverse to the direction of propagation of the beam being
calculated such that:

$$A \leq \lambda / \delta\theta$$

where λ is the wavelength of the incident radiation, and

$\delta\theta$ is the optical resolution of the apparatus used in implementing

25 the method.

4. The method of any preceding claim, wherein the analyser is rotated in
incremental steps α :

$$\alpha \leq \delta\theta/2$$

30 where $\delta\theta$ is the optical resolution of the apparatus used in implementing
the method.

5. The method of any preceding claim, including the use of a PIN diode
detector to detect the radiation reflected from the analyser.

6. The method of any preceding claim, wherein the radiation is produced using a characteristic line source.

5

7. The method of claim 6, wherein the characteristic line source is a rotating anode source.

10 8. The method of any preceding claim, including the step of calculating a complex scattering amplitude of the irradiated portion of the object from the detected intensities, and including the step of determining a complex scattering function of the irradiated portion by taking an inverse Fourier Transform of the complex scattering amplitude.

15 9. The method of claim 8, including the steps of:
normalising the detected intensities;
calculating the modulus of the complex scattering amplitude from the normalised intensity;
calculating phase information for the complex scattering amplitude
20 from the modulus of the complex scattering amplitude; and
determining the complex scattering amplitude from the modulus and phase information.

25 10. The method of claim 2, 8 or 9, including the step of determining a complex refractive index profile of the irradiated portion of the object from the complex scattering function.

30 11. Apparatus for the analysis of an object, the apparatus including:
a source of non-planar penetrating radiation;
a monochromator for diffracting the non-planar penetrating radiation to provide a beam of monochromatic penetrating radiation;
a detector for detecting radiation that passes through the object;
an analyser for diffracting radiation that passes through the object onto the detector;

means for rotating the analyser between a plurality of angular positions;
and

means for recording the intensity of the radiation incident on the detector
as a function of analyser position.

5

12. The apparatus of claim 11, including means for determining a complex
scattering function of the portion of the object under analysis from the intensity
measurements.

10

13. The apparatus of claim 11 or 12, including a slit member defining a slit
through which the radiation beam passes prior to the beam's incidence on the
object, the slit size A in a direction transverse to the direction of propagation of
the beam being such that:

$$A \leq \lambda/\delta\theta$$

15

where λ is the wavelength of the incident radiation, and
 $\delta\theta$ is the optical resolution of the apparatus.

14. The apparatus of claim 11, 12 or 13, wherein the analyser is rotated in
incremental steps α :

20

$$\alpha \leq \delta\theta/2$$

where $\delta\theta$ is the optical resolution of the apparatus.

15. The apparatus of any of claims 11 to 14, wherein the detector comprises
a PIN diode detector.

25

16. The apparatus of any of claims 11 to 15, wherein the radiation source is
a characteristic line source.

30

17. The apparatus of claim 16, wherein the radiation source is a rotating
anode source.

18. The apparatus of claim 12, wherein the means for determining the
complex scattering function includes means for calculating a complex scattering
amplitude of the irradiated portion of the object from the detected intensities,

and means for determining a complex scattering function of the irradiated portion by taking an inverse Fourier Transform of the complex scattering amplitude.

- 5 19. The apparatus of claim 18, including:
means for normalising the detected intensities;
means for calculating the modulus of the complex scattering
amplitude from the normalised intensity;
means for calculating phase information for the complex scattering
10 amplitude from the modulus of the complex scattering amplitude; and
means for determining the complex scattering amplitude from the
modulus and phase information.

- 15 20. A method of analysis of an object, the method including the steps of:
generating penetrating radiation;
diffracting the radiation from a monochromator to provide a beam of
monochromatic penetrating radiation;
passing the beam of radiation through a slit, the slit size A (in a direction
transverse to the direction of propagation of the beam) being calculated such
20 that:

$$A \leq \lambda / \delta\theta$$

- where λ is the wavelength of the incident radiation, and
 $\delta\theta$ is the optical resolution of the apparatus used in implementing
the method;
25 irradiating a portion of the object with the beam;
diffracting radiation that passes through the object onto a detector from
an analyser;
rotating the analyser through a plurality of angular positions; and
measuring the intensity of the radiation incident on the detector as a
30 function of analyser position.

21. The method of claim 20, including the step of determining a complex scattering function of the portion of the object under analysis from the intensity measurements.

22. The method of claim 20 or 21, wherein the penetrating radiation is non-planar penetrating radiation.

- 5 23. The method of any of claims 20 to 22, wherein the analyser is rotated in incremental steps α :

$$\alpha \leq \delta\theta/2$$

where $\delta\theta$ is the optical resolution of the apparatus used in implementing the method.

10

24. The method of any of claims 20 to 23, including the use of a PIN diode detector to detect the radiation reflected from the analyser.

- 15 25. The method of any of claims 20 to 24, wherein the radiation is produced using a characteristic line source.

26. The method of claim 25, wherein the characteristic line source is a rotating anode source.

- 20 27. The method of any one of claims 20 to 26, including the step of calculating a complex scattering amplitude of the irradiated portion of the object from the detected intensities, and including the step of determining a complex scattering function of the irradiated portion by taking an inverse Fourier Transform of the complex scattering amplitude.

25

28. The method of claim 27, including the steps of:

normalising the detected intensities;

calculating the modulus of the complex scattering amplitude from the normalised intensity;

- 30 calculating phase information of the complex scattering amplitude from the modulus of the complex scattering amplitude; and

determining the complex scattering amplitude from the modulus and phase information.

29. The method of claim 21, 27 or 28, including the step of determining a complex refractive index profile of the irradiated portion of the object from the complex scattering function.

5 30. Apparatus for the analysis of an object, the apparatus including:
a source of penetrating radiation;
a monochromator for diffracting the penetrating radiation to provide a
beam of monochromatic penetrating radiation;
a slit member defining a slit through which the beam passes prior to the
10 beam's incidence on the object, the slit size A in a direction transverse to the
direction of propagation of the beam being such that:

$$A \leq \lambda / \delta\theta$$

where λ is the wavelength of the incident radiation, and

$\delta\theta$ is the optical resolution of the apparatus;

15 a detector for detecting radiation that passes through the object;
an analyser for diffracting radiation that passes through the object onto
the detector;

means for rotating the analyser between a plurality of angular positions;

and

20 means for recording the intensity of the radiation incident on the detector
as a function of analyser position.

31. The apparatus of claim 30, including means for determining a complex
scattering function of the portion of the object under analysis from the intensity
25 measurements.

32. The apparatus of claim 30 or 31, including a slit member defining a slit
through which the radiation beam passes prior to the beam's incidence on the
object, the slit size A in a direction transverse to the direction of propagation of
30 the beam being such that:

$$A \leq \lambda / \delta\theta$$

where λ is the wavelength of the incident radiation, and

$\delta\theta$ is the optical resolution of the apparatus.

33. The apparatus of claim 30, 31 or 32, wherein the analyser is rotated in incremental steps α :

$$\alpha \leq \delta\theta/2$$

where $\delta\theta$ is the optical resolution of the apparatus.

5

34. The apparatus of any of claims 30 to 33, wherein the detector comprises a PIN diode detector.

10

35. The apparatus of any of claims 30 to 34, wherein the radiation source is a characteristic line source.

36. The apparatus of claim 35, wherein the radiation source is a rotating anode source.

15

37. The apparatus of claim 31, wherein the means for determining the complex scattering function includes means for calculating a complex scattering amplitude of the irradiated portion of the object from the detected intensities, and means for determining a complex scattering function of the irradiated portion by taking an inverse Fourier Transform of the complex scattering amplitude.

20

38. The apparatus of claim 37, including:

means for normalising the detected intensities;

means for calculating the modulus of the complex scattering

25

amplitude from the normalised intensity;

means for calculating phase information for the complex scattering amplitude from the modulus of the complex scattering amplitude; and

means for determining the complex scattering amplitude from the modulus and phase information.

30

39. A method of analysis of an object, the method including the steps of:

irradiating a portion of the object with a beam of monochromatic x-rays;

detecting the intensity profile of an angular spectrum of the x-rays

emerging from the irradiated portion; and

determining a complex scattering function for the irradiated portion of the object under analysis.

40. A method of analysis of an object, the method including the steps of:

5 irradiating a portion of the object with a beam of monochromatic x-ray radiation;

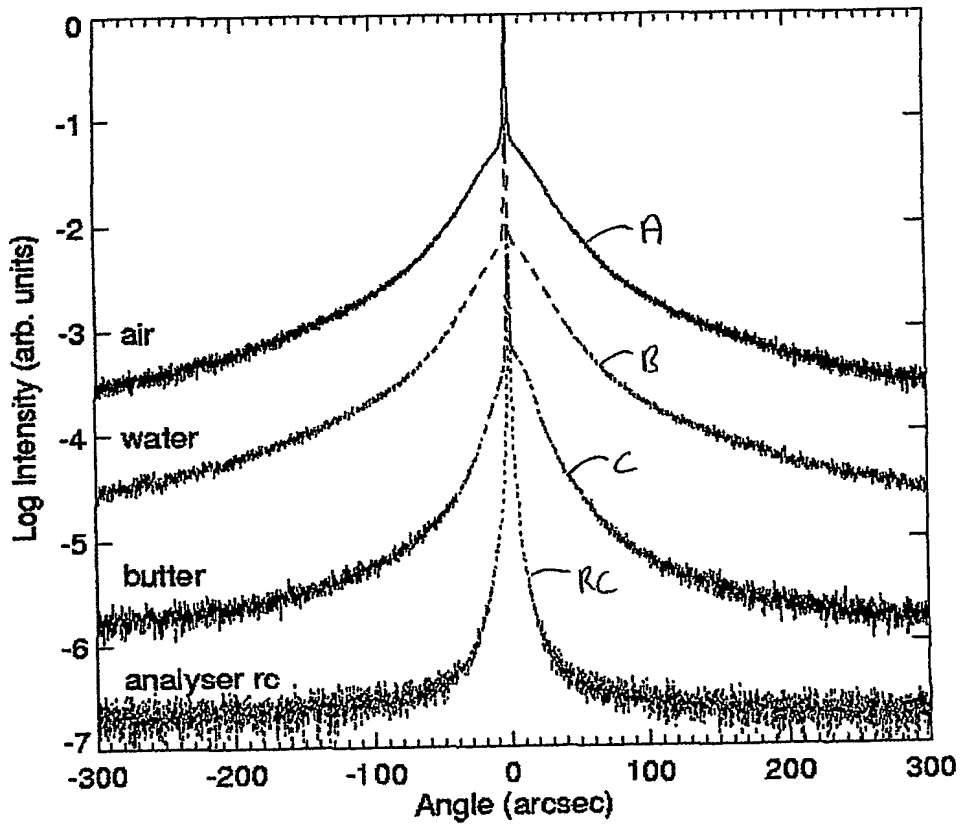
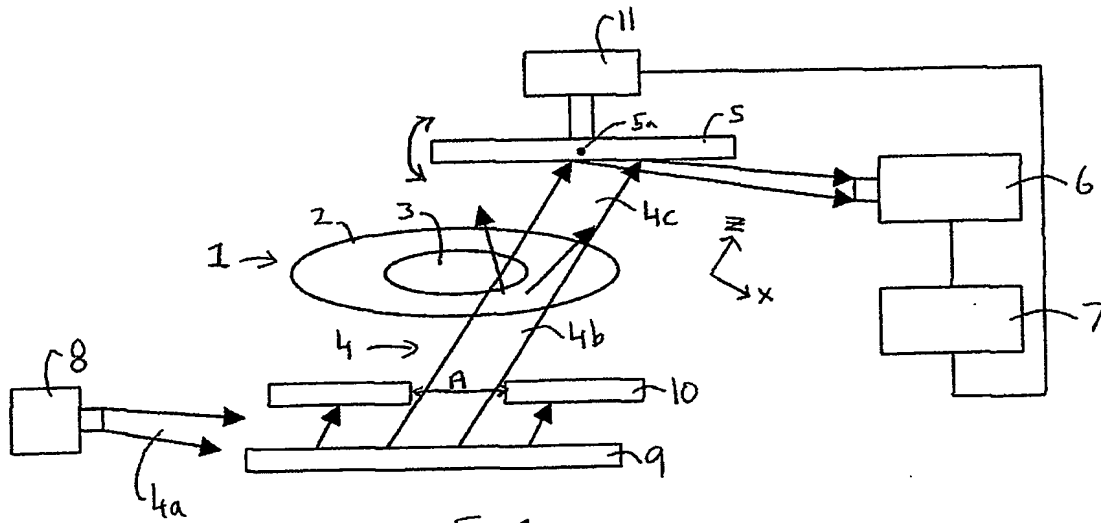
diffracting x-rays emerging from the sample into an x-ray detector using an analyser means; and

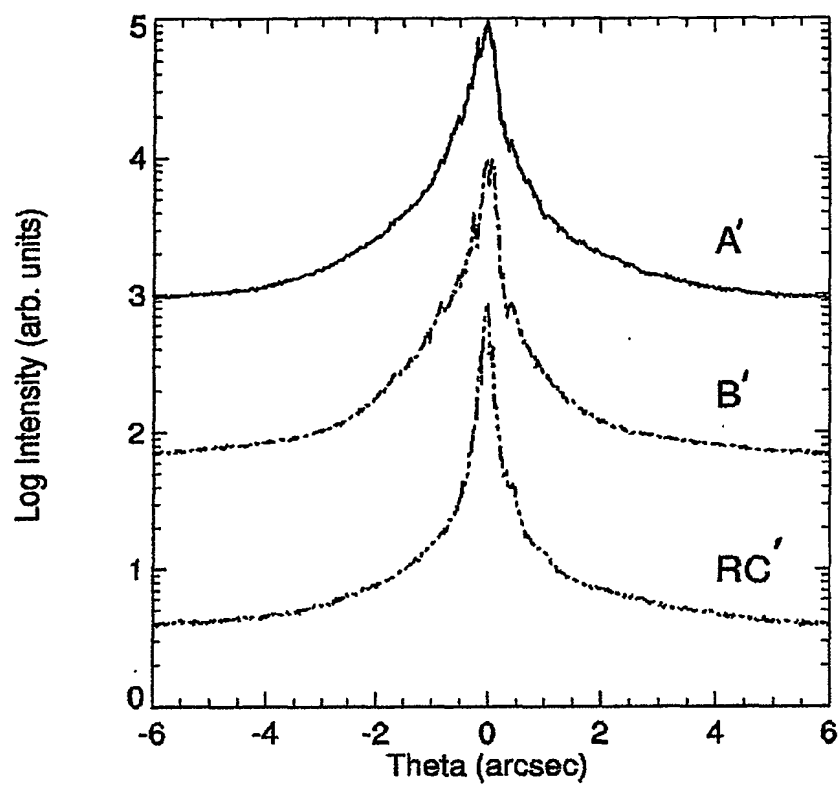
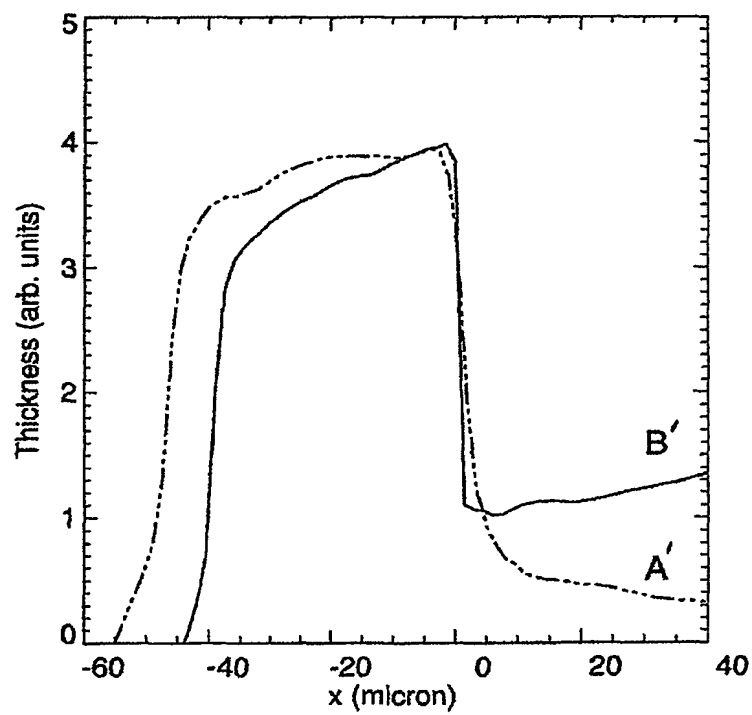
10 obtaining an angular spectrum of non-Bragg diffracted x-ray intensities as a function of angular position of the analyser means.

41. A method of analysis of an object, the method including the step of

collecting generic x-ray diffraction data from a portion of the object and

15 analysing the data to obtain a complex refractive index of the sampled portion in a direction transverse to the beam propagation.



Fig. 3Fig. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU03/00748

A. CLASSIFICATION OF SUBJECT MATTER												
Int. Cl. ⁷ : G01N 23/02												
According to International Patent Classification (IPC) or to both national classification and IPC												
B. FIELDS SEARCHED												
Minimum documentation searched (classification system followed by classification symbols)												
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched												
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) DWPI, JAPIO IPC G01N 23/IC with KEYWORDS : (monochromat+ or ((one or single) (w) (waveglenth or frequency)) or ((silicon or Si) (w) crystal))												
C. DOCUMENTS CONSIDERED TO BE RELEVANT												
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.										
X	US 5987095 A (CHAPMAN et al.) 16 November 1999 Abstract; Fig. 1; Col. 4, lines 32 - col. 5, lines 61	1,2,5-7,11,12, 15-17,39,40										
X	US 5319694 A.(INGAL et al.) 7 Jun 1994 Abstract; col. 4, lines 7-23; col. 5, lines 54-63; Fig. 3	1,2,5-7,11,12, 15-17,39,40										
X	WO 01/79823 A (CHAPMAN et al.) 25 October 2001 Abstract; page 6, lines 14-23; page 15, line 20 - page 17, line 9; Fig. 1	1,2,5-7,11,12, 15-17,39,40										
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex												
<p>* Special categories of cited documents:</p> <table border="0"> <tr> <td>"A" document defining the general state of the art which is not considered to be of particular relevance</td> <td>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</td> </tr> <tr> <td>"E" earlier application or patent but published on or after the international filing date</td> <td>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</td> </tr> <tr> <td>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</td> <td>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</td> </tr> <tr> <td>"O" document referring to an oral disclosure, use, exhibition or other means</td> <td>"&" document member of the same patent family</td> </tr> <tr> <td>"P" document published prior to the international filing date but later than the priority date claimed</td> <td></td> </tr> </table>			"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone	"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art	"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family	"P" document published prior to the international filing date but later than the priority date claimed	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention											
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone											
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art											
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family											
"P" document published prior to the international filing date but later than the priority date claimed												
Date of the actual completion of the international search 28 July 2003		Date of mailing of the international search report 05 SEP 2003										
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustalia.gov.au Facsimile No. (02) 6285 3929		Authorized officer LARS KOCH Telephone No : (02) 6283 2551										

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/00748

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 95/05725 A (COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION) 23 February 1995 Abstract; page 4, line 20 - page 5, line 1; page 6, lines 9-17; page 12, line 23 - page 13, line 4; Figs 1,4	1,2,5-7,11,12,15-17,39,40

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/00748

Box I Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos :
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos :
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos :
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box II Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See Supplemental Sheet.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:
1-40

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU03/00748

Supplemental Box

(To be used when the space in any of Boxes I to VIII is not sufficient)

Continuation of Box No: II

The international application does not comply with the requirements of unity of invention because it does not relate to one invention or a group of inventions so linked as to form a single inventive concept. In coming to this conclusion the International Searching Authority has found that there are two inventions:

1. Claims 1-10, 20-29, 39, 40 are directed to a method for analysis of an object comprising the following steps :
irradiating the object with a beam of monochromatic X-rays,
diffracting the X-rays emerging from the object,
obtaining an angular spectrum of the diffracted X-ray intensities.
An apparatus for performing the above method is also claimed in claims 11-19, 30-38.
It is considered that the steps of irradiating the object with a beam of monochromatic X-rays, diffracting the emerging X-rays from the object and obtaining an angular spectrum of diffracted X-ray intensities comprise a first "special technical feature" (for the method claims).
2. Claim 41 is directed to a method of analysis of an object including the steps of :
collecting generic X-ray diffraction data from a portion of the object,
analysing the data to obtain a complex refraction index of the sampled portion in a direction transverse to the beam propagation.
It is considered that the steps of collecting generic X-ray diffraction data from a portion of the object and analysing the data to obtain a complex refraction index of a sampled portion comprise a second "special technical feature".

Since the above mentioned group of claims do not share any of the technical features identified, a "technical relationship" between the inventions, as defined in PCT rule 13.2 does not exist. Accordingly the international application does not relate to one invention or to a single inventive concept.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU03/00748

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member	
US 5987095	WO 9816817		
US 5319694	EP 539608	US 5579363	WO 9221016
WO 200179823	AU 200153566	US 2002027970	
WO 9505725	AU 74538/94	US 5802137	US 5850425
END OF ANNEX			